



Serial No.: 10/719,598
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s)	:	Joel D. Oxman et al.
Serial No.	:	10/719,598
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Title	:	Photoinitiator Systems with Anthracene-Based Electron Donors for Curing Cationically Polymerizable Resins
Group/Art Unit	:	1711
Examiner	:	Susan W. Berman
Confirmation No.	:	3112
Atty. Docket No.	:	800528-0014
Assignee:		Curators Of The University Of Missouri University

DECLARATION OF JOEL D. OXMAN

Mail Stop AF
Commissioner for Patents
PO Box 1450
Alexandria VA 22313-1450

Dear Madam:

I hereby declare as follows:

1. My name is Joel D. Oxman. I have been employed since 1983 by the 3M Company, one of the assignees of the above-captioned invention and I am currently a Division Scientist. During this period, I have supervised and/or participated in many of the experiments described in the above-captioned application.
2. In addition to being a co-inventor for the above-caption application, I am also a co-inventor on over 50 issued patents.
3. I have carefully reviewed the prior art references cited by the Examiner in the July 12, 2006 Final Office Action.

Certificate of Mailing Under 37 C.F.R. 1.8

I hereby certify that this correspondence is being deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to: Mail Stop AF, Commissioner for Patents, PO Box 1450, Alexandria, VA 22313-1450, on:

Date:

November 13, 2006

Signature:

Mary E. Menard

Printed Name:

Mary E. Menard

The Director is hereby authorized to charge any additional amount required, or credit any overpayment, to Deposit Account No. 19-4409.

4. While at 3M, I worked with co-inventor Craig Dykstra, a postdoctoral fellow from the University of Kansas City Missouri from approximately February 2002 through August 2003. During that time period, Dr. Dykstra performed many experiments on various photoinitiator systems, such as the Depth of Cure experiments described herein. I also provided significant guidance and direction for these efforts. More specifically, I was routinely and directly involved with the interpretation and understanding of Dr. Dykstra's experimental design and results. In making this Declaration, I have carefully reviewed and summarized the data recorded by Dr. Dykstra in notebooks and reports.
5. The following experimental data shows a surprising synergistic results for the visible light curing characteristics of a cationically curable epoxy composition comprising an iodonium salt (Tolylcumyliodonium Tetrakis(pentafluorophenyl) Borate (Rhodorsil 2074)), a visible light ketone sensitizer (camphorquinone (CPQ)) and an alkoxy anthracene electron donor (2-ethyl-9,10-dimethoxy anthracene (EDMO)) as compared to similar compositions in the absence of either CPQ or EDMO.
6. **Preparation of Polymerizable Compositions.** The following polymerizable compositions were prepared by combining the following materials in to a sealed vial and mixing until homogeneous:

Stock Solution 1 (SL1)

Ingredient	<u>parts by weight(gm)</u>	<u>weight per cent</u>
Cygepsi (Silorane epoxy material)	20.00	94.25
Poly THF (Polytetrahydrofuran)	0.62	2.92
Rhodorsil 2074 (iodonium salt)	0.60	2.83

SL1-EDMO Preparation of Epoxy Resin Composition with EDMO

Ingredient	<u>parts by weight (gm)</u>	<u>weight per cent</u>
SL1	5.010	99.90
EDMO	0.005	0.10

Stock Solution 2 (SL2) with CPQ

Ingredient	<u>parts by weight (gm)</u>	<u>weight per cent</u>
Cygepsi (Silorane epoxy material)	74.70	93.34
Poly THF (Polytetrahydrofuran)	2.53	3.16
Rhodorsil 2074 (iodonium salt)	2.40	3.00
CPQ (camphorquinone)	0.40	0.50

SL2-EC Preparation of Epoxy Resin Composition with EDMO and CPQ

Ingredient	<u>parts by weight (gm)</u>	<u>weight per cent</u>
SL2	5.010	99.90
EDMO	0.005	0.10

7. **Preparation of Filled Compositions.** The resin compositions SL1-EDMO, SL2 and SL2 were converted to filled compositions by the addition of 70% by weight of a silane

treated quartz filler as described in the patent application as "Filler A" The resulting filled compositions were viscous translucent pastes that are further characterized and labeled below:

F1

<u>Ingredient</u>	<u>parts by weight (gm)</u>	<u>weight per cent</u>
SL1-EDMO (contains EDMO)	1.50	30.0
Filler A	3.50	70.0

F2

<u>Ingredient</u>	<u>parts by weight (gm)</u>	<u>weight per cent</u>
SL2 (contains CPQ)	1.50	30.0
Filler A	3.50	70.0

F3

<u>Ingredient</u>	<u>parts by weight (gm)</u>	<u>weight per cent</u>
SL2-EC (contains CPQ and EDMO)	1.50	30.0
Filler A	3.50	70.0

8. **Sample Evaluation.** Samples were evaluated for depth of cure via light activation as a means of determining the relative effectiveness of the various photoinitiator combinations. Filled compositions F1-F3 were evaluated according to "Depth of Cure Test Method A " of the current patent application. Paste samples were transferred into a cylindrical opaque plastic mold (11-mm deep, 4-mm diameter) and irradiated for 40 seconds with a 900 mW/cm² curing light that provides visible light between approximately 400-500nm XL3000, 3M ESPE Company). The cured sample was removed from the mold and the uncured paste was scraped off the sample with a plastic applicator approximately within one minute after the light exposure. The length of the sample measured in mm with a micrometer. Duplicate samples were performed for each filled composition
9. **Results.** Set out below in Table I are depths of cure results for the samples containing EDMO, CPQ and CPQ+EDMO

Table I Depth of Cure of 70 % Filled Epoxy Compositions with Various Photoinitiator Combinations Irradiated for 40 seconds with a 900 mW/cm² curing light

Sample	Camphorquinone concentration in Unfilled Epoxy	EDMOA Concentration in Unfilled Epoxy	Approximate Cure depth of Filled Epoxy Resin(mm)
F1	0.00%	0.10%	3.5 mm
F2	0.50%	0.00%	4.8 mm
F3	0.50%	0.10%	11.0 mm

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10. **Conclusions.** The data shown in Table I clearly illustrates that in a cationically curable epoxy system that the combination of a visible light ketone sensitizer (such as camphorquinone (CPQ)) in combination with an alkoxy substituted anthracene donor (such as EDMO) provides a dramatically enhanced depth of cure when compared to analogous compositions with only the visible light ketone sensitizer (CPQ) or the alkoxy-substituted anthracene (EDMO) individually when exposed to the identical visible light source. I believe that the significant synergistic nature of this combination is surprising and not obvious based on the prior art.
11. I hereby declare that all statements made herein are of my own knowledge or based on information and believed to be true, and that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both (18 U.S.C. § 1001) and may jeopardize the validity of the patent application or any patent issuing thereon.

Date: 11/10/06Signature: Joel D. Oxman
Joel D. Oxman